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Charge Transfer Excitons in Bulk Heterojunctions of a Polyfluorene Copolymer and a Fullerene Derivative**

By Maria Antonietta Loi,* Stefano Toffanin, Michele Muccini, Michael Forster, Ulrich Scherf, and Markus Scharber

The photophysical properties of blends of fluorene copolymer and the fullerene derivative PCBM are analyzed with a particular attention to photovoltaic applications. The properties of the blends are determined by the relative alignment of the HOMO energy levels. In the blend where the HOMO levels of the copolymer and the fullerene are aligned there is not signature of charge stabilization and photovoltaic effect. While in the blend where there is an offset between the HOMO levels the charge stabilization is accompanied by good photovoltaic performances. The photoluminescence spectrum of the latter blend is characterized by the appearance of a new peak at low energy with a lifetime of a few ns that red-shifts with the increase of the PCBM percentage. The feature is attributed to the emission from a charge-transfer exciton that is red-shifted by the change of dielectric constant of the medium.

1. Introduction

Photovoltaic devices based on three-dimensional heterostructures (bulk heterojunction) composed by a light adsorbing and hole conducting polymer and a fullerene derivative (electron conducting) have been an interesting research topic for more than 10 years.^[1] With the recent achievements in power conversion efficiency (3–5%)^[2,3] it is now clear that this class of solar cells can also have an industrial future. Nevertheless, several questions about the functioning mechanism are pending and several problems have to be solved to bring them to be of real market interest.

Serious limitations to the performances of the devices at the moment are: the relatively low charge carrier mobility in the polymer (that range around 10^{-4} cm² V⁻¹ s⁻¹) and the generally narrow absorption in the low energy part of the visible solar spectrum.

The mechanism of the charge separation, even if has been the subject of extensive studies in literature^[4] is still to be fully understood. For example, it is not yet completely clear why in some blends (as is for MDMO-PPV/PCBM^[5]) the transport properties could be improved with respect to the pristine polymer upon addition of a fullerene derivative. The desire to shed light on this and other interesting phenomena is the reason for the growing interest in the photophysical properties of such blends. In fact, photophysical studies can be extremely helpful to understand if there is an intermediate state between the excitons formed upon light absorption and the “free” charges. And if an intermediate state exists, it is in the ground state like a charge transfer exciton^[6] or is it only in the excited state as an exciplex^[7]? Moreover, what is the lifetime of the intermediate excited state^[8]?

Recently, great efforts have been put in finding photoactive materials having absorption spectra with better overlap with the solar spectrum.^[9] Polyfluorene copolymers in this respect offer the possibility to have good mobilities ($>10^{-4}$ cm² V⁻¹ s⁻¹) and to tune the gap towards low energy by copolymerization with low-bandgap monomers. In addition it is possible to use alternating monomers with donor–acceptor functions. This approach has been rather successful in the case of copolymers of fluorene and benzothiadiazole. In fact, solar cells using as active layer bulk heterojunction of this family of copolymers and the soluble fullerene derivative PCBM showed lately very promising performances.^[10,11] In our studies we used the symmetric substituted alternate copolymer poly((2,7-(9,9-(di-*n*-octyl)fluorene)-*alt*-5,5-(4',7'-di-thienyl-2',1',3'-benzothiadiazole)) (F8DTBT), whose structure is closely related to that of poly((2,7-(9,9-(di-*n*-octyl)fluorene)-*alt*-(2,1,3-benzothiadiazole)) (F8BT) that has previously shown excellent properties in light emitting devices^[12] and photovoltaic activity in polymer–polymer blends.^[13]

Herein we report a study of the photophysical properties of F8DTBT/PCBM and F8BT/PCBM blends with particular at-

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tention to photovoltaic applications. The blend F8DTBT/PCBM shows promising performances as active layer in photovoltaic devices, while F8BT/PCBM shows a very low photocurrent. Both copolymers upon mixing with PCBM show a quite efficient quenching of the photoluminescence, but only the blend of F8DTBT/PCBM shows evidences of charge separation and of photocurrent generation. The possibility of an energy transfer between the F8BT and PCBM will be analyzed.

The photoluminescence spectrum of the blend containing F8DTBT shows the appearance of a new peak at low energy with a lifetime of few ns. Such a new PL feature is ascribed to the emission of a charge transfer exciton formed upon the PCBM blending. The low energy peak shows a strong red-shift with the increase of the PCBM percentage in the blend that we attribute to the variation of dielectric constant of the medium. The determination of the nature of the intermediate excited state can be extremely important for both the understanding of the conduction mechanism of the device and the improvement of the device performances.

2. Results and Discussion

In Figure 1 the chemical structure of two copolymers of polyfluorene, namely poly((2,7-(9,9-(di-*n*-octyl)fluorene)-*alt*-5,5-(4',7'-di-thienyl-2',1',3'-benzothiadiazole)) (F8DTBT) and poly((2,7-(9,9-(di-*n*-octyl)fluorene)-*alt*-(2,1,3-benzothiadiazole)) (F8BT) are shown together with the structure of the soluble fullerene derivative [6,6]-phenyl-C61-butyric acid methyl ester (PCBM) that is the most used acceptor in plastic solar cells devices.^[14] The HOMO–LUMO energy levels of the molecules under investigation are reported in Figure 1.

In Figure 2a and b are reported the current–voltage characteristics and the external quantum efficiency, respectively, of

the photovoltaic cells having as active layer F8DTBT/PCBM and F8BT/PCBM. The device fabricated with the F8DTBT blend shows interesting performances, in particular, the short-circuit current is $\sim 4 \text{ mA cm}^{-2}$, open-circuit voltage is 800 mV; the fill factor is ~ 0.4 for a 20 mm^2 device. It should be noted that the device shown above have not been optimized, which may explain the low performance compared to efficiencies presented earlier.^[10,11,15] Devices made with F8BT/PCBM blend as active layer have extremely poor performances. Due to the chemical similarities of the two polymers such a great difference at a first glance is unexpected.

In Figure 3 the absorption spectra of F8DTBT, PCBM and of blend films are reported. The absorption of F8DTBT has two main peaks at $\sim 390 \text{ nm}$ and $\sim 560 \text{ nm}$, PCBM spectra shows a peak at $\sim 335 \text{ nm}$ and then degrade smoothly in the visible with a pronounced shoulder. The polymer/PCBM blends shows more complex shapes where can be identified the main peaks of the component materials but with the intensity that is not in agreement with a linear combination of the absorptions in the nominal percentages of the film. To have a better correspondence of the calculated and experimental spectra it is necessary to underestimate the percentage of PCBM in the blend. However, even with the underestimation of the PCBM concentration the experimental data are not easily reproduced with a linear superposition of the neat films spectra. Such a discrepancy could be ascribed both to the morphology of the film and to the interaction of the two moieties in the blend, i.e., ground state interactions. However, it is difficult to demonstrate the latter effect with transmission measurements due to expected low oscillator strength.

In Figure 4 the absorption spectra of F8BT, PCBM and of the blends with the same percentages used for the other copolymer (F8DTBT) blends are reported.

The absorption of F8BT shows two main features, the first one at $\sim 320 \text{ nm}$ that has a shoulder at $\sim 338 \text{ nm}$ and the second one with the maximum at 463 nm . All the features of the absorption of the blended films can be reproduced by a weighted linear combination of F8BT and PCBM absorption in agreement with the composition of the film (see Fig. 4). The small differences shown by the measured blend spectra with respect to the calculated ones can be ascribed to small underestimation of the percentage of the copolymer in the blend or, more probably, to the dispersion of one species in the other that decrease the solid state effect. It is noteworthy that in the case of F8BT/PCBM there are no huge discrepancies between the measured spectrum of the blend and the calculated linear superposition of the absorption of the components as reported for the F8DTBT blends (Fig. 3).

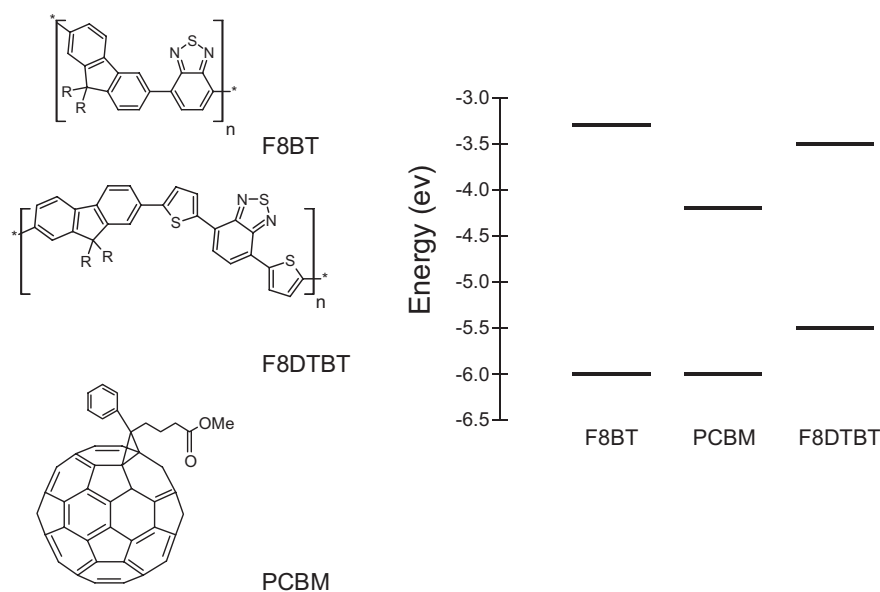


Figure 1. Left: Chemical structures of F8BT, F8DTBT and PCBM. R is C_8H_{17} , Me is CH_3 . Right: HOMO–LUMO energy levels of the three molecules as obtained by electrochemical measurements.

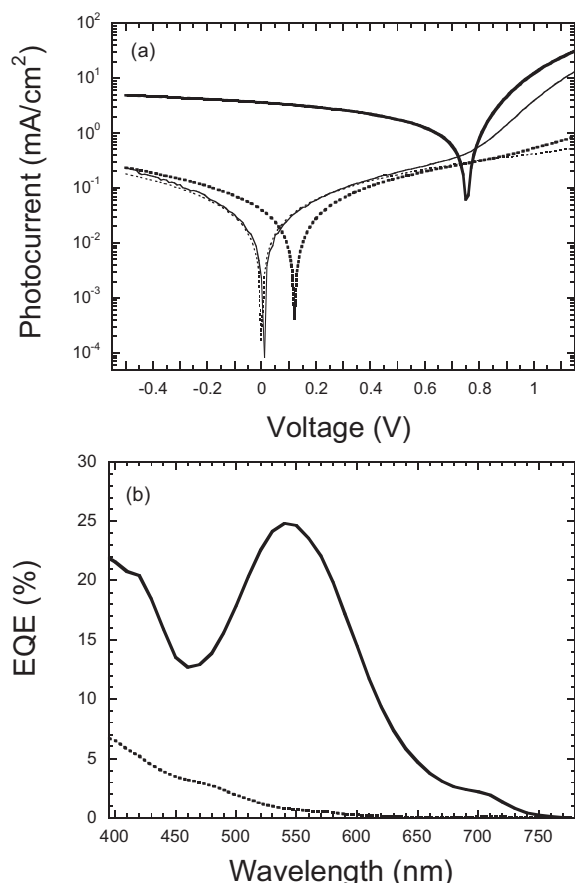


Figure 2. a) Semilogarithmic plot of the current density-voltage characteristics of a F8DTBT/PCBM (thick lines) and F8BT/PCBM (thin lines) devices. Dark (dashed lines) and 100 mWcm⁻² white light illumination curves (continuous lines) are reported. b) EQE measurements of the F8DTBT/PCBM (continuous line) and F8BT/PCBM (dashed line) devices.

The correlation of steady state photoluminescence spectroscopy and time resolved measurements on bulk heterojunctions could be extremely informative on the nature of the photo excitations. In particular, it can allow the detection of the existence and identification of the nature of the elusive intermediate state between the exciton and the free charges.

In Figure 5 the normalized photoluminescence spectra of the F8DTBT and PCBM neat films and of the 5 %, 20 %, 33 %, and 66 %PCBM blends are reported. Upon blending the copolymer with PCBM at first (5 %PCBM) a broadening of the PL it is shown, then, with 20 % of PCBM in the blend there is the appearance of a new feature in the spectra at ~714 nm (the main peak of F8DTBT is at 700 nm) and of a shoulder blue shifted with respect to the original position of the main PL peak of the copolymer. A further increase of the PCBM quantity in the blend gives rise to a very broad emission with a main peak ~785 nm for the 33 %PCBM and at ~822 nm for the 66 %PCBM blend. It is important to note that none of these wavelengths correspond to the emission of the neat PCBM that is quite narrow with a maximum at 735 nm and a shoulder at ~802 nm (Fig. 5). The blending of the copolymer with PCBM gives rise to a quenching of the PL over ~240 times (integrated

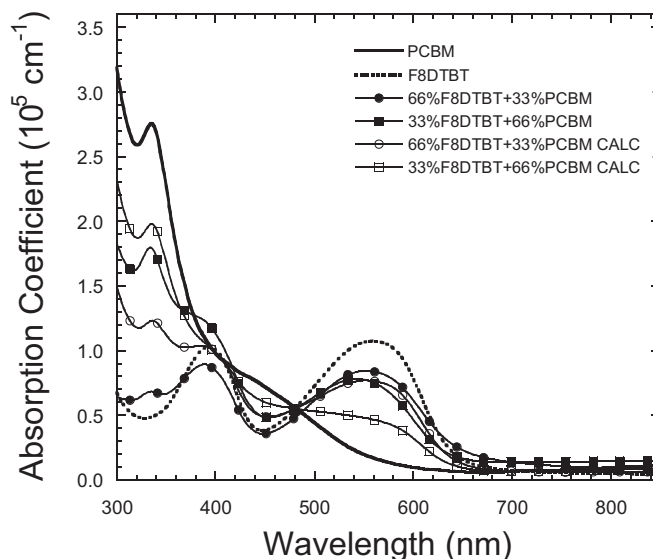


Figure 3. Absorption spectra of thin films of F8DTBT, PCBM and F8DTBT/PCBM blends in proportion of 1:3 and 3:1. The absorption spectra calculated as superposition of the measured absorption of the two components in the proportion of the blend are indicated by empty squares (33 %PCBM) and empty circles (66 % PCBM).

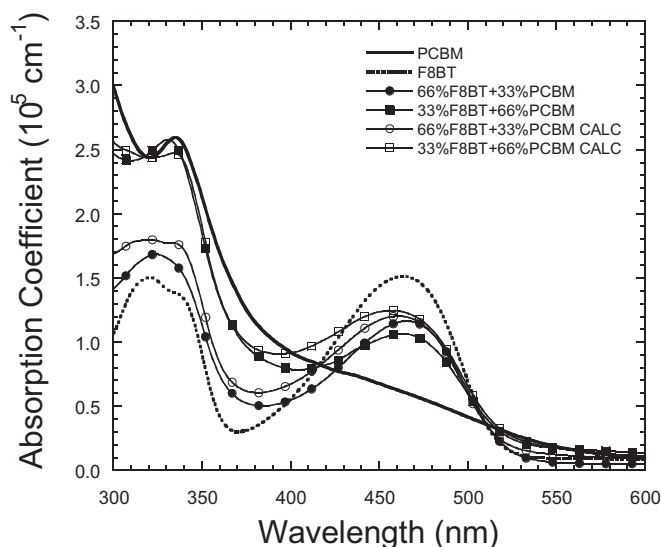


Figure 4. Absorption spectra of thin films of F8BT, PCBM and F8BT/PCBM blends in proportion of 3:1 and 1:3. The absorption spectra calculated as superposition of the measured absorption of the two components in the proportion of the blend are indicated by empty squares (33 %PCBM) and circles (66 % PCBM).

over all the spectral range) from the neat copolymer film to the 66 %PCBM blend and an even stronger decrease at the wavelength corresponding at the maximum emission of F8DTBT.

It is noteworthy that in the blends with higher PCBM content there is no signature of the PL fingerprint of both F8DTBT and PCBM. Moreover, the emission in the near infrared has the interesting peculiarity to red shift by increasing the amount of PCBM in the blend. A new excited state could be the origin of this new feature. Among the possible excited

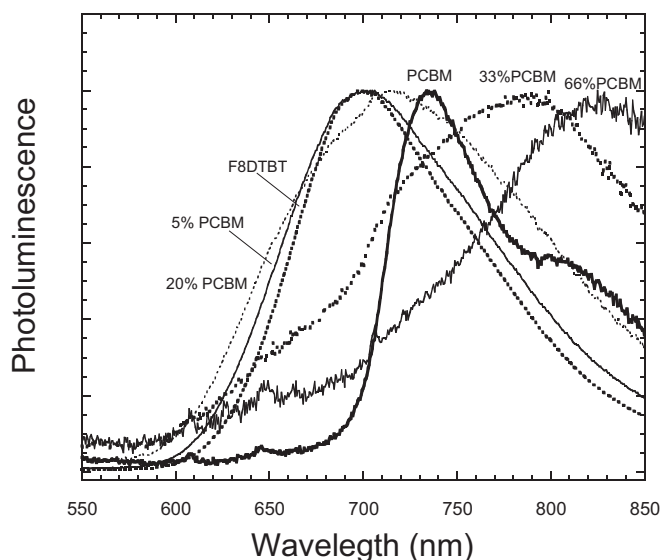


Figure 5. Photoluminescence spectra of F8DTBT, PCBM and F8DTBT/PCBM-blend thin films. Different blends are reported: 5%, 20%, 33%, 66%PCBM. The spectra are normalized to the maximum intensity.

states there are charge transfer excitons, exciplexes and triplet excitons.

Charge transfer exciton (CTE) are correlated electron-hole states where the electron is located in a neighbor molecule of the one where the hole is located, optical transitions from the ground state to the CTE are possible even if the oscillator strength is general weak.^[16] In the case of exciplexes the ground state is dissociative, consequently, they can be revealed only with fluorescence measurements.^[16] It is important that the decay times in the case of the exciplexes are generally rather long (up to 40 ns) as are normally long the decay times of triplet excitons.^[12,17]

In Figure 6 the photoluminescence dynamics for the neat F8DTBT and PCBM films and for the 66%PCBM blend are reported. At wavelengths corresponding to the polymer maximum emission (630 nm) there is a strong reduction of the lifetime in the 66%PCBM blend (limited by the instrumental resolution) with respect to the lifetimes of the neat copolymer film ($\tau_1=0$ ps; $\tau_2=330$ ps). The new low energy peak (820 nm), revealed in the steady state PL spectra, shows a bi-exponential decay with $\tau_1=320$ ps and $\tau_2=3.1$ ns. Such a long decay time is neither found in the pristine polymer nor in the PCBM neat film in agreement with the fact that the new emission originates from a different excited species. The measured lifetimes are more compatible with an emission from CTE.

Evidences of emission from a charge transfer exciton recombination have been found in blends of fullerenes and poly(vinylcarbazole),^[18] while the shift of the charge transfer PL peak with the increasing of the fullerene concentration was not reported. Such a shift can be explained with the decrease of the binding energy of the charge transfer exciton.

Charge transfer excitons (CTE) are less bounded species compared to Frenkel excitons and can be treated as weakly interacting electron-hole pairs,^[16] which have optical transitions that can be resembled to Rydberg transition in a hydrogen

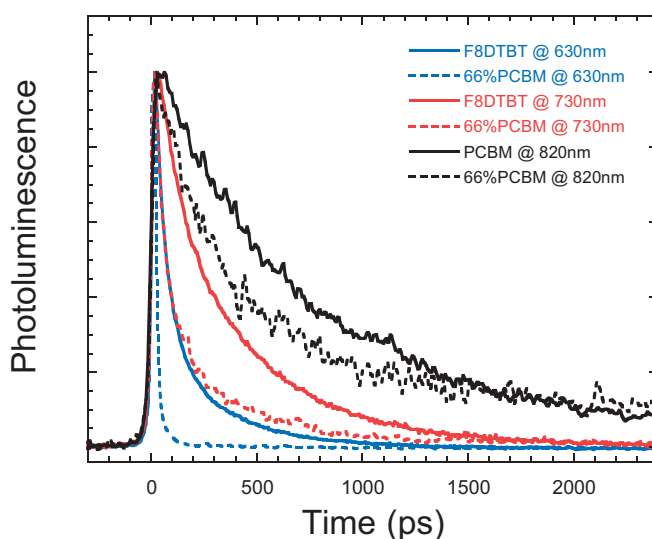


Figure 6. Photoluminescence dynamics of F8DTBT, PCBM and of 33%F8DTBT/66%PCBM blend at different wavelengths. The intensity is normalized at $T=0$.

atom. Consequently, the increasing of the dielectric constant of the medium due to the increased concentration of PCBM ($\epsilon(\text{PCBM}) \sim 3.9$; $\epsilon(\text{Polymer}) \sim 2.5-3.0$)^[19] will result in a less bounded CTE; in agreement with the increased red shift of the emission with the increase of the PCBM percentage. Moreover, it is important to underline that the energy levels of F8DTBT and PCBM do allow the formation of charge transfer excitons.

Recently, evidences of weak CTE have been reported for blends of MDMO-PPV/PCBM,^[6] and the presence of a CTE have been considered a possible explanation of the increase of the hole mobility of 2 orders of magnitude in MDMO-PPV/PCBM blend with respect to the neat MDMO-PPV.^[5] The presence of CTE in the active layer of a photovoltaic device could explain an improvement of the charge transport in blends^[20] and help understanding the solar cells electrical characteristics.

In Figure 7 the photoinduced absorption of F8DTBT and F8DTBT/PCBM are plotted. The appearance of a new absorption in the spectrum of the blend at 1.7 eV is in agreement with the presence of stabilized charges in the polymer.

The photophysics of the blend F8BT/PCBM was found to be completely different from the one of the F8DTBT blend. In Figure 8 the normalized PL spectra of F8BT and PCBM neat films and of the blends containing 5, 20, 33, and 66% of PCBM are reported. The photoluminescence of the blends is composed by the superposition of the PL of the two component materials. The integrated intensity decrease of a factor 30 from the neat F8BT film to the 66%PCBM film, with an almost complete quenching of the PL at the wavelength of the maximum PL of the copolymer. The PL spectra of the blends with high PCBM content have the same spectral shape than the one of the neat PCBM film with a small shift towards the blue for the film with the lower content of PCBM, which can be ascribed to the dispersion of the PCBM molecules in the polymer matrix.

In Figure 9 the PL decays of the F8BT and PCBM neat films are compared with the one of the 66%PCBM blend. As shown

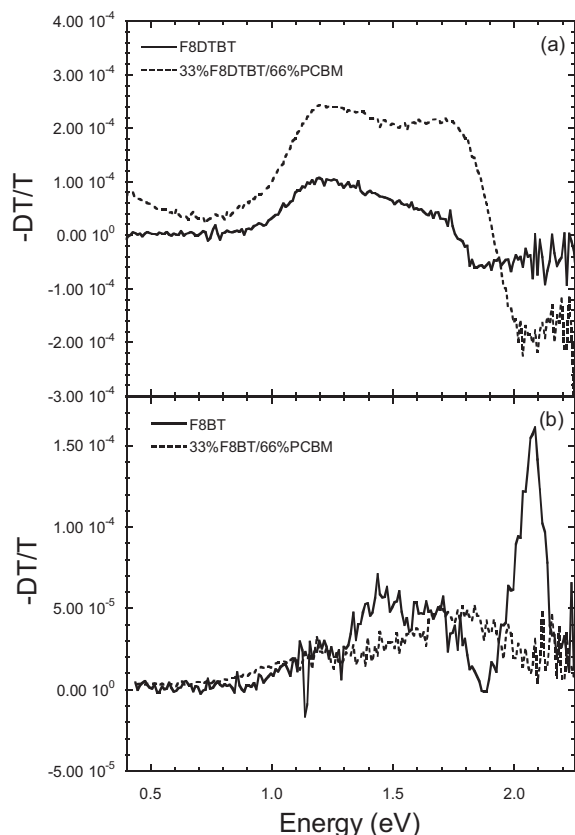


Figure 7. a) Photoinduced absorption spectra of F8DTBT and 33 %F8BT/66 %PCBM blend. b) Photoinduced absorption spectra of F8BT and 33 %F8BT/66 %PCBM blend. Excitation wavelength 488 nm.

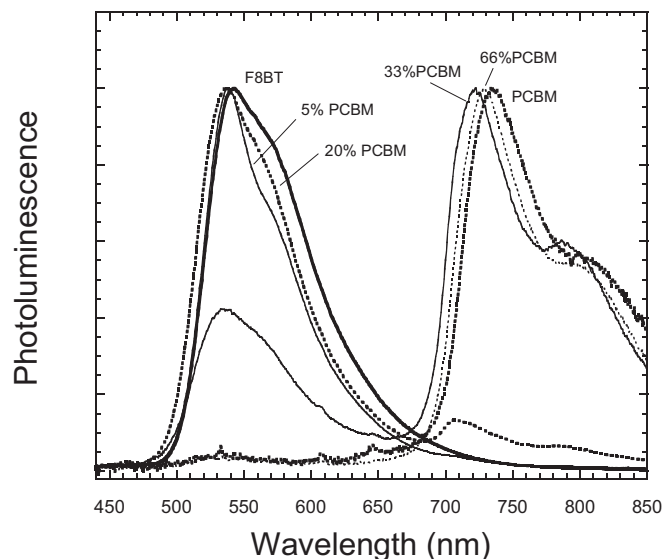


Figure 8. Photoluminescence spectra of F8BT, PCBM and F8BT/PCBM-blend thin films. Different blends are reported: 5 %, 20 %, 33 %, 66 %PCBM. The spectra are normalized to the maximum intensity, each spectrum with respect to its most intense peak.

in Figure 8 the PL spectra of the blend F8BT/PCBM is composed by the F8BT PL component (530 nm) that shows an ex-

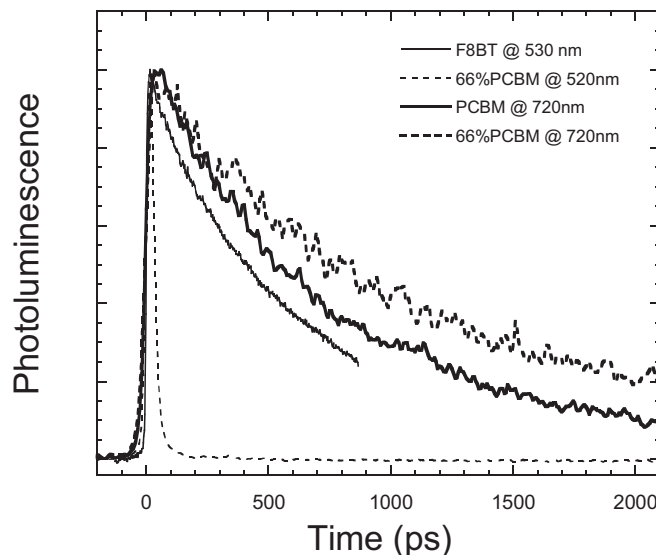


Figure 9. Photoluminescence dynamics of F8BT, PCBM and of 33 %F8BT/66 %PCBM blend at different wavelengths. The intensity at $T=0$ is normalized.

tremely short lifetime, comparable with the resolution of the experimental set-up. The PCBM component (720 nm) in the blend shows a longer lifetime ($\tau_1=670$ ps and $\tau_2=1.8$ ns) with respect to the one of the neat PCBM film ($\tau_1=350$ ps and $\tau_2=1.0$ ns) that can be explained with the dispersion of the PCBM molecules in the copolymer matrix.

The quenching of the F8BT photoluminescence can not be ascribed to the charge separation as the photoinduced absorption (PIA) spectrum (Fig. 7b) of the blend does not show any signature of localized charges in the polymer. A possible motivation for the copolymer PL quenching could be the energy transfer between F8BT and PCBM.

On the one hand, the alignment of HOMO levels of the PCBM and F8BT at about -6 eV (see Fig. 1) could favor the energy transfer from the copolymer to the PCBM. On the other hand, it is important to note that, eventually, energy transfer would be rather efficient even at low concentration of PCBM, at the opposite the photoluminescence measurements show that the emission signal at low PCBM concentrations is low and increases with the increasing of the PCBM percentage. Another possible explanation could be the transfer of the excitation to triplet state of PCBM, but also in this case the PIA measurement (Fig. 7b) does not fully support this explanation. In fact, comparing the PIA spectrum of the blend with that of the pristine polymer a very weak signature at 1.8 eV appears and another weak feature at 1.45 eV (probably due to the triplet state) disappears. The surprisingly low signal does not allows a clear understanding of the physics of this blend, probably because the phenomena involved are too fast for the time resolution of this technique (in the reported experiment it is possible to detect only species with lifetime longer than 6 ns). A very recent report^[21] analyzes similar experimental data as-signing the quenching of F8BT to the energy transfer to the singlet state of the PCBM then transferred to the triplet state.

Such explanation even if plausible is not in agreement with our measurements of PL rise time of several ps both for the blend sample and pure PCBM.

3. Conclusions

The photophysical properties of blends of fluorene copolymer and PCBM are determined by the HOMO energy level of the copolymer. In F8BT/PCBM blend the quenching of the photoluminescence does not lead to charge stabilization while the bulk heterojunction composite made of F8DTBT and PCBM leads to a charge stabilization and is a promising active layer for the next generation of plastic solar cells. Experimental evidences of the presence in the blend of an excited state intermediate between the exciton and the free carriers are found. The photoluminescence spectrum of the blend containing F8DTBT shows the appearance of a new peak at low energy with a lifetime of a few ns. This new PL feature is ascribed to the emission of a charge transfer excitons formed upon PCBM blending. The low energy peak shows a strong red-shift with the increase of the PCBM percentage in the blend that we ascribe to the change of dielectric constant of the medium. The presence of CTE in the bulk heterojunction can be extremely important both for the understanding of the conduction mechanism of the device and for the improvement of the device performances.

4. Experimental

In all described experiments ortho-dichloro-benzene (ODCB) was used as solvent. Typically 10 mg of polymer were dissolved in 1 mL of solvent and PCBM was added to achieve the desired polymer fullerene ratio (in wt %). For optical experiments 100 nm thin polymer layers were deposited by blade-coating. Deposition parameters were chosen in a way to achieve comparable layer thicknesses for different blend concentrations. Different blend films with PCBM percentages from 5 % to 66 % have been prepared and investigated.

Photovoltaic devices were built on indium tin oxide (ITO) coated glass. The substrate was covered with a thin film of PEDOT:PSS (Baytron PH from Bayer AG) by blade-coating. On top of it the active layers (polymer–fullerene ratio 1:2 (wt %)) were deposited by blade coating. The deposition parameters were varied to achieve thin (100 nm) homogenous layers. The top electrodes were vapor deposited through a shadow mask, using few angstrom of LiF and 100 nm of Al. The device fabrication and characterization was performed under Ar atmosphere. For external quantum efficiency (EQE) measurements devices were sealed using a UV-curable epoxy resin and thin glass plate.

I/*V* characteristics were measured by a Keithley 2400 SMU unit under illumination intensity of 100 mWcm^{−2} of white light from an ORIEL Solar Simulator. A lock-in technique was used to measure the EQE spectra. With this technique the electrons produced from the cell under short-circuit conditions are related to the number of incident photons. Light intensity correction was performed using a calibrated Si photodiode.

Photoluminescence (PL) spectra were obtained exciting with the second harmonic of a femtosecond Ti:Sapphire laser at 400 nm and 460 nm, no relevant differences between the two excitations have been found. Time resolved measurements were performed with a monochromator coupled to a Hamamatsu streak camera (time resolution ~2 ps). PL spectra were recorded with an optical multichannel analyser. All photoluminescence measurements were performed with the samples

kept in vacuum (~10^{−6} mbar) at room temperature. The decay times are obtained by fitting the experimental data with bi-exponential functions $A \cdot \exp(t/\tau_1) + B \cdot \exp(t/\tau_2)$, where A and B are the weights accounting for the $t = 0$ intensity.

Photoinduced absorption (PIA) spectra were measured using a mechanically modulated (170 Hz) Ar-ion laser beam at 488 nm as a pump. The changes in the white light (tungsten-halogen lamp) probe beam transmission ($-\Delta T$) due to the pump beam, were detected after dispersion with a monochromator in the range from ~0.5 to 2.4 eV with a Si-InGaAsSb sandwich detector and recorded phase sensitively with a dual-phase lock-in amplifier. The probe light transmission (T) was recorded separately using the same chopper frequency. The PIA spectra ($-\Delta T/T$) are obtained after correction for the sample luminescence and normalization on the probe light transmission. PIA measurements were carried out on sample at 100 K, the signal was not measurable at room temperature.

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